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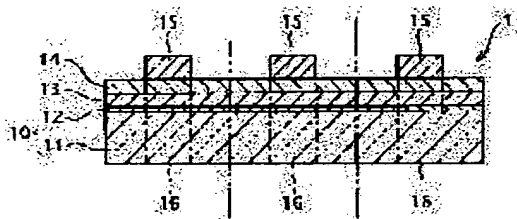
(54) VAPOR DEPOSITION METHOD AND ORGANIC ELECTROLUMINESCENCE ELEMENT

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a vapor deposition method by which contamination by gas, moisture, etc., can be surely reduced in an vapor deposition method by which an organic layer is film-formed by using an organic vapor deposition material and to provide an organic

electroluminescence(EL) element of which excellent quality can be secured by reducing contamination with impurities.

SOLUTION: When organic layers 13, 14 are formed by vapor deposition by using the organic vapor deposition material, the organic vapor deposition material of pellet-shape in which gas content is made to be ≤ 1 mol.% is used. Thereby, the contamination by gas, moisture, etc., can be surely reduced and when the organic EL element 1 is manufactured, the organic EL element 1 having a long life, low driving voltage and high quality can be obtained.



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CLAIMS

[Claim(s)]

[Claim 1] It is the vacuum evaporation method which is the vacuum evaporation method which forms an organic layer using an organic vacuum evaporation material, and is characterized by forming the aforementioned organic vacuum evaporation material in the shape of a pellet, and making gas content into less than [1mol%].

[Claim 2] It is the vacuum evaporation method characterized by carrying out deaeration processing by heating the aforementioned organic vacuum evaporation material in a vacuum in the vacuum evaporation method indicated to the claim 1.

[Claim 3] It is the organic electroluminescent element characterized by being the organic electroluminescent element equipped with the electrode and organic layer which were formed on the substrate, forming the aforementioned organic layer by carrying out the vacuum evaporation of the organic vacuum evaporation material on the aforementioned substrate, and forming the aforementioned organic vacuum evaporation material in the shape of a pellet, and making gas content into less than [1mol%].

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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the organic EL element equipped with the vacuum evaporation method which forms an organic layer using an organic vacuum evaporation material, the electrode formed on the substrate, and the organic layer in detail about the vacuum evaporation method and an organic EL element (organic electroluminescent element).

[0002]

Background of the Invention] In recent years, the organic EL element which is a luminescence device containing an organic layer attracts attention, and research is advanced towards the use to a display etc. Generally this organic EL element is equipped with organic layers, such as an anode plate (transparent electrode) and a luminous layer, and the structure which carried out the laminating of the cathode (counterelectrode) on the substrate. On the other hand, in the electrophotography copying machine of the Carlsson method, the organic photo conductor equipped with the photosensitive layer which consists of the organic substance as an electrophotography photo conductor is used. Membrane formation of the organic layer in these organic EL elements, electrophotography photo conductors, etc. is performed by the vacuum deposition method which an organic vacuum evaporation material which consists of the organic substance is heated [vacuum deposition method], and evaporates it. The powdered thing which carried out melting deaeration and ground the organic substance of a raw material is used for this organic vacuum evaporation material.

[0003] However, in the organic substance's tending to adsorb the gas which is an impurity at the time of the trituration etc., since an organic powdered vacuum evaporation material has the large touch area with the atmosphere, it is easy to adsorb impurities, such as moisture. At the time of the vacuum evaporation of an organic layer, gas, moisture, etc. which were adsorbed evaporate with an organic vacuum evaporation material, and it mixes in an organic layer or they adhere to a substrate etc. Because of contamination by impurities, such as such gas, the target function may not fully be obtained in an organic EL element, an electrophotography photo conductor, etc. By using what carried out pressing of the organic substance powder, and was used as the tablet as what solves such a problem as an organic vacuum evaporation material, the method (JP,5-204196,A) of suppressing evaporation of the gas at the time of vacuum evaporation and an organic vacuum evaporation material made to evaporate are introduced into a cold trap, and after making an impurity condense and removing, the method (JP,50-3973,A) of using for vacuum evaporation etc. is proposed.

[0004]

[Problem(s) to be Solved by the Invention] By the method of tablet-izing organic substance powder, since it is only only carrying out pressing of the organic substance powder, gas, the organic solvent especially incorporated during the crystal of the organic substance cannot be removed, but there is a problem that contamination by gas cannot fully be reduced out of vacuum evaporation material. On the other hand, by the method of introducing into a cold trap, although the nonvolatile component of the impurities in an organic vacuum evaporation material is removable, since it cannot fully remove

volatile components, such as gas, it cannot avoid contamination by gas etc. Moreover, if an organic EL element is created by these methods, since contamination of a luminous layer, a substrate, etc. cannot fully be suppressed, element performance degradation and the problem that a life becomes short especially arise.

[0005] The purpose of this invention is in the vacuum evaporation method which forms an organic layer using an organic vacuum evaporation material to offer the vacuum evaporation method that contamination by impurities, such as gas and moisture, can be reduced certainly. Moreover, other purposes of this invention are in the organic EL element equipped with the electrode and organic layer which were formed on the substrate to offer the organic EL element which can secure the quality which was excellent by reducing contamination by the impurity.

[0006]

[Means for Solving the Problem] Although it was based also on the property of the gas contained in an organic vacuum evaporation material as a result of this invention persons' inquiring wholeheartedly in order to solve the aforementioned technical problem, when the gas beyond 5mol% was contained in an organic vacuum evaporation material, degasifying occurred from an organic vacuum evaporation material at the time of vacuum evaporation, and the knowledge that an organic layer, a substrate, etc. were polluted by this gas was acquired. In addition, gas here is matter which is below the vacuum evaporation temperature grade of an organic vacuum evaporation material, and evaporates, and the matter which evaporates at temperature higher than the temperature near vacuum evaporation temperature is not included. this invention is restricting the amount of the gas in an organic vacuum evaporation material, and tends to attain the aforementioned purpose.

[0007] Specifically, this invention is the vacuum evaporation method which forms an organic layer using an organic vacuum evaporation material, and the aforementioned organic vacuum evaporation material is characterized by being formed in the shape of a pellet, and making gas content into less than [1mol%].

[0008] Here, gas means the matter which evaporates among the impurities in an organic vacuum evaporation material at the temperature below near the temperature at the time of the vacuum evaporation of an organic vacuum evaporation material among matter other than an organic vacuum evaporation material which carries out vacuum evaporation that is, irrespective of the organic substance and an inorganic substance. What it is heated not only at a gaseous thing but at the time of vacuum evaporation, and is evaporated in the atmosphere (ordinary temperature) is contained in this gas.

[0009] In this invention, since gas content of an organic vacuum evaporation material is made into less than [1mol%] and the amount of the gas as an impurity which evaporates at the time of the vacuum evaporation of an organic layer can be lessened sharply, contamination by gas can be reduced certainly. Moreover, rather than a powdered thing, since it is formed in the shape of a pellet, since an organic vacuum evaporation material can make area in contact with the atmosphere small, adsorption of the moisture which is an impurity is made as for it to the minimum. Therefore, since evaporation of the moisture from organic substance vacuum evaporation material etc. is made to the minimum, contamination by impurities, such as moisture, can be reduced certainly. Moreover, since heat conduction of itself becomes good unlike powder, when a lot of material is taught, vacuum evaporation conditions are stable for a short time.

[0010] As for the aforementioned organic vacuum evaporation material, it is above desirable by heating in a vacuum to carry out deaeration processing. Thus, if the organic substance is heated in a vacuum, since gas, such as an organic solvent incorporated during the crystal of the organic substance, is also removable and it can deaerate efficiently, the gas content in an organic vacuum evaporation material can be reduced certainly.

[0011] Furthermore, the gas content of the aforementioned organic vacuum evaporation material is less than [0.1mol%] more preferably, and gas content has a desirable method of a low. Moreover, in the case of vacuum evaporation, since gas occurs from the vacuum tub itself, effect sufficient also by using the low organic vacuum evaporation material of gas content rather than this on the basis of the gas

which occurs from this vacuum tub is acquired. The method of presuming from change of the degree of vacuum in the case of deaeration processing as a measuring method of gas content, the gas-chromatography method, the method of searching for from the weight reduction before and behind deaeration processing, etc. are employable. Moreover, although not limited, since it has a possibility of becoming at the time of vacuum evaporation and decomposing into an elevated temperature when especially the kind of organic vacuum evaporation material has too large molecular weight, the smaller one of molecular weight is desirable, for example, it is 2000 or less molecular weight.

[0012] Moreover, although especially the configuration or size of a pellet are not limited, what it is easy to create is desirable. As a configuration of a pellet, the shape of a globular shape and a pillar, a rectangular parallelepiped configuration, etc. are employable, for example. Moreover, as for the size of a pellet, it is desirable that an overall diameter sets to 0.1-100mm.

[0013] On the other hand, this invention is the organic electroluminescent element equipped with the electrode and organic layer which were formed on the substrate, and it is characterized by forming the aforementioned organic layer by carrying out the vacuum evaporation of the organic vacuum evaporation material on the aforementioned substrate, and forming the aforementioned organic vacuum evaporation material in the shape of a pellet, and making gas content into less than [1mol%].

[0014] In this invention, since the thing of the shape of a pellet which made gas content less than [1mol%] is used as an organic vacuum evaporation material, gas, moisture, etc. which are an impurity in an organic vacuum evaporation material can be lessened and contamination of an organic layer or a substrate can be reduced certainly, the low quality organic EL element of driver voltage with a long life is obtained.

[0015] By the type in which the aforementioned organic EL element considers the substrate side as the ejection side of light, the thing of following the (1) - (4) is mentioned for the order of a laminating on a substrate front face as an example of the lamination of an organic EL element.

(1) An anode plate / luminous layer / cathode (2) anode plate / luminous layer / electron-injection layer / cathode (3) anode plate / hole-injection layer / luminous layer / cathode (4) anode plate / hole-injection layer / luminous layer / electron-injection layer / cathode [0016] Here, usually, although a luminous layer is formed of a kind or two or more sorts of organic luminescent material, it may be formed of mixture with an organic luminescent material, hole-injection material, and/or electron-injection material. Moreover, the protective layer for preventing permeation of the moisture to an element on it, as the element concerned is covered on the periphery of the element of lamination mentioned above may be prepared.

[0017] When making a substrate side into the ejection side of light, the aforementioned substrate consists of matter which gives high permeability (in general 80% or more) to luminescence (EL light) from an organic EL element at least, and, specifically, the tabular object, sheet-like object, or film-like object which consists of transparent glass, transparent plastics, a quartz, etc. can be used for it.

[0018] As a material of an anode plate, cathode, a luminous layer, a hole-injection layer, an electron-injection layer, and a protective layer, a well-known material can be used conventionally, respectively. For example, as an anode material, a metal with a large (for example, 4eV or more) work function, an alloy, electrical conductivity compounds, or such mixture are used preferably. As an example, it is metals, such as gold and nickel, and CuI, ITO and SnO₂. Dielectric transparent materials, such as ZnO, etc. are mentioned. Especially, the point of productivity or a controllability to ITO is desirable. Although the thickness of an anode plate is based also on material, it is usually selectable suitably within the limits of 10nm - 1 micrometer.

[0019] Moreover, as a cathode material, the small (for example, 4eV or less) metal of a work function, an alloy, electrical conductivity compounds, or such mixture are used preferably. As an example, rare earth metal, such as the alloy of sodium, a sodium potassium alloy, magnesium, a lithium, magnesium, and silver or a mixed metal, aluminum, aluminum/aluminum 2O₃, an indium, and an ytterbium, etc. is mentioned. Although the thickness of cathode is based also on material, it is usually selectable suitably within the limits of 10nm - 1 micrometer. Also in any of an anode plate and cathode, below hundreds of ohms / ** of the sheet resistance are desirable. In addition, the size of the work function made into

criteria in case an anode material and a cathode material are chosen is not limited to 4eV.

[0020] An organic vacuum evaporation material (organic luminescent material) of the luminous layer which is an organic layer While being able to pour in an electron hole from an anode plate or a hole-injection layer at the time of the luminous layer for organic EL elements, i.e., electric-field impression The pouring function in which an electron can be poured in from cathode or an electron-injection layer, What is necessary is just to be able to form the layer which has the transportation function to which the poured-in charge (an electron and an electron hole at least on the other hand) is moved by the force of electric field, the luminescence function to offer the place of the reunion of an electron and an electron hole and to tie this to luminescence, etc. As the example, a benzothiazole system, a benzimidazole system, The fluorescent brightener and metal chelate-ized oxy-NOIDO compound of systems, such as a benzo oxazole system A styryl benzenoid compound, a JISUCHIRIRU pyrazine derivative, a polyphenyl system compound, 12-phthaloperi non, 1, 4-diphenyl-1,3-butadiene, 1, 1, 4, and 4-tetraphenyl-1,3-butadiene, a NAFUTARU imide derivative, The metal complex of a perylene derivative, an OKISA diazole derivative, an aldazine derivative, a peeler JIRIN derivative, a cyclopentadiene derivative, a pyrrolo pyrrole derivative, a styryl amine derivative, a coumarin system compound, an aromatic dimethyldiyne compound, and an eight-quinolinol derivative etc. is mentioned. Although especially the thickness of a luminous layer is not limited, it is usually suitably chosen within the limits of 5nm - 5 micrometers.

[0021] An organic vacuum evaporation material (hole-injection material) of the hole-injection layer as an organic layer should just have the pouring nature of an electron hole, or electronic obstruction nature. As the example, a triazole derivative, an OKISA diazole derivative, An imidazole derivative, the poly aryl alkane derivative, a pyrazoline derivative, A pyrazolone derivative, a phenylenediamine derivative, an arylamine derivative, An amino substitution chalcone derivative, an oxazole derivative, a styryl anthracene derivative, full -- me -- non -- a derivative, a hydrazone derivative, a stilbene derivative, and a silazane derivative -- Conductive polymer oligomer, such as a polysilane system compound, an aniline system copolymer, and thiophene oligomer, a porphyrin compound, an aromatic tertiary-amine compound, a styryl amine compound, an aromatic JIMECHIRI DIN system compound, etc. are mentioned. Although especially hole-injection layer thickness is not limited, either, it is usually suitably chosen within the limits of 5nm - 5 micrometers. A hole-injection layer may be two or more layer structure which consists of a kind of the material mentioned above, or two sorts or more and which may be structure much more and consists of two or more layers of the same composition or different-species composition.

[0022] The electron-injection layer which is an organic layer that what is necessary is just to have the function to transmit the electron poured in from cathode to a luminous layer as an example of the material (electron-injection material) nitration full -- me -- non -- a derivative, an anthra quinodimethan derivative, and a diphenyl quinone derivative -- Heterocycle tetracarboxylic acid anhydrides, such as a thiopyran dioxide derivative and a naphthalene perylene, A carbodiimide, a deflection ORENIRIDEN methane derivative, an anthra quinodimethan derivative, The metal complex of an anthrone derivative, an OKISA diazole derivative, and an eight-quinolinol derivative, a metal free phthalocyanine and a metal phthalocyanine or the thing by which these ends are replaced with the alkyl group, the sulfone machine, etc., a JISUCHIRIRU pyrazine derivative, etc. are mentioned. Although especially electron-injection layer thickness is not limited, either, it is usually suitably chosen within the limits of 5nm - 5 micrometers. An electron-injection layer may be two or more layer structure which consists of a kind of the material mentioned above, or two sorts or more and which may be structure much more and consists of two or more layers of the same composition or different-species composition.

[0023] And the copolymer which is made to carry out copolymerization of the monomer mixture containing at least a tetrafluoroethylene and a kind of comonomer, and is obtained as an example of the material of a protective layer, The fluorine-containing copolymer which has a cyclic structure in a copolymerization principal chain, polyethylene, polypropylene, A polymethylmethacrylate, a polyimide, a polyurea, poly tetrapod FUROORO ethylene, A polychlorotrifluoroethylene resin, poly dichlorodifluoroethene, The copolymer of a chlorotrifluoroethylene and dichlorodifluoroethene, The

absorptivity matter of 1% or more of water absorptions, and the dampproof matter of 0.1% or less of water absorptions, Metals, such as In, Sn, Pb, Au, Cu, Ag, aluminum, Ti, and nickel, MgO, SiO, SiO₂ aluminum₂ O₃ GeO, NiO, CaO, BaO, and Fe₂ O₃ Y₂ O₃ TiO₂ etc. -- a metallic oxide and MgF₂ LiF, AlF₃, and CaF₂ etc. -- a metal fluoride etc. is mentioned

[0024] Moreover, it is not limited especially about the formation method of each class (an anode plate and cathode are included) except the organic layer which constitutes an organic EL element, either. As the formation methods, such as an anode plate and cathode, to the pan which can apply a vacuum deposition method, the spin coat method, the cast method, the sputtering method, the LB method, etc., for example about a protective layer A vacuum deposition method, the spin coat method, the sputtering method, the cast method, the MBE (molecular beam epitaxy) method, The cluster ion beam method, the ion play DINGU method, a plasma polymerization method (the RF excited ion play DINGU method), a reactive-sputtering method, a plasma CVD method, laser CVD, heat CVD, gas source CVD, etc. are employable.

[0025] Although the formation method of each class except an organic layer can be suitably changed according to the material to be used, when aiming at simplification of a facility, and shortening of the production time since an organic EL element can be formed only by this vacuum deposition method if a vacuum deposition method is used in formation of each class which constitutes an organic EL element, it is advantageous.

[0026] In addition, the configuration and size of each class (a protective layer is included) which constitute an organic EL element are not necessarily the same. Moreover, when plane view of the element is carried out, all other layers are not surely settled on the electrode formed in right above [of a substrate].

[0027]

[Embodiments of the Invention] Hereafter, one gestalt of operation of this invention is explained based on a drawing.

<Composition of an organic EL element> Organic EL element 1 of this operation gestalt is shown in drawing 1. This organic EL element 1 is equipped with a substrate 11, the anode plate (transparent electrode) 12 which consists of an ITO film formed on this substrate 11, the hole-injection layer 13 as an organic layer and luminous layer 14 which were formed on this anode plate 12, and these organic layers 13 and the cathode (counterelectrode) 15 formed on 14.

[0028] A substrate 11 is a rectangle plate-like glass substrate, and the band-like anode plate 12 is formed by the one side along with the longitudinal direction. On the hole-injection layer 13 by which the laminating was carried out on this anode plate 12, and the luminous layer 14, three band-like cathode 15 prolonged in the direction which intersects perpendicularly with the longitudinal direction of a substrate 11, i.e., the direction which intersects perpendicularly with an anode plate 12, is formed. In organic EL element 1 of this operation gestalt, when plane view is carried out, an anode plate 12 and cathode 15 lap, and the luminescence side 16 of a flat-surface configuration rectangle is formed in the field to which the hole-injection layer 13 and the luminous layer 14 were infixed among these electrodes 12 and 15.

[0029] The hole-injection layer 13 which is an organic layer among such organic EL elements 1 is formed by carrying out the vacuum evaporation of the organic vacuum evaporation material for the hole-injection layer 13 concerned on the anode plate 12 of a substrate 11. The luminous layer 14 which is an organic layer is formed like this by carrying out the vacuum evaporation of the organic vacuum evaporation material for the luminous layer 14 concerned on the hole-injection layer 13 of a substrate 11. Such organic vacuum evaporation material is formed in the shape of a pellet, and the gas content is made into less than [1mol%].

[0030] <Production of an organic vacuum evaporation material> Each organic vacuum evaporation material which constitutes the hole-injection layer 13 and a luminous layer 14 is produced in the respectively following procedures. With this operation gestalt, deaeration processing and fabrication of the organic substance are performed by heating the powdered organic substance in a vacuum. That is, organic substance powder is put into the container made from molybdenum, and it sets in the vacuum tub of a vacuum evaporation system. In addition, this container has two or more crevices of the

configuration corresponding to the pellet, and puts the organic substance powder of the specified quantity into these crevices, respectively.

[0031] Subsequently, if a vacuum tub is exhausted with a vacuum pump and a vacuum tub becomes a predetermined degree of vacuum, a container will be heated so that it may become predetermined temperature. In addition, what is necessary is just to measure the degree of vacuum of a vacuum tub using an ionization vacuum gage etc. If a container is heated, while the organic substance powder in the crevice will fuse, the gas as an impurity contained in the organic substance is emitted, and, thereby, the degree of vacuum of a vacuum tub falls. If it continues heating a container to predetermined temperature as it is, the gas in organic substance powder will decrease, the evaporation will decrease, and, thereby, the degree of vacuum of a vacuum tub will become high. And if the gas contained in organic substance powder is almost exhausted, the degree of vacuum of a vacuum tub will return to the aforementioned predetermined degree of vacuum (degree of vacuum before heating). The gas in organic substance powder is removed by such deaeration processing. Gas, such as an organic solvent incorporated during the crystal of the organic substance, is certainly removable by heating organic substance powder especially. The heating temperature of the container at this time is set as the temperature which is a grade in which the organic substance fuses and does not evaporate, and the heating time of a container is set up more than the time taken to recover the degree of vacuum which fell by generating of gas to a predetermined degree of vacuum (degree of vacuum before heating).

[0032] After returning to the state before the degree of vacuum of a vacuum tub heating, heating of a container is ended, the fused organic substance is cooled, and since the configuration where the crevice of a container was imitated is solidified, it takes out from a vacuum tub. Thereby, the pellet which is an organic vacuum evaporation material is obtained. Thus, organic substance powder is heated in a vacuum, and if it deaerates until it returns to the state before the degree of vacuum of a vacuum tub heating, the gas content of the pellet obtained will become less than [1mol%]. Moreover, since a pellet becomes smaller than the time of the surface area in contact with the atmosphere being in a powder state, absorption of the moisture after deaeration processing etc. can be suppressed to the minimum.

[0033] <Production of an organic EL element> With this operation gestalt, the hole-injection layer 13, a luminous layer 14, and cathode 15 are formed one by one by the vacuum deposition method to the substrate 11 with anode plate 12. That is, after preparing the transparent support substrate 10 which formed the ITO film 12 beforehand on a substrate 11 and washing this transparent support substrate 10, it fixes to the substrate electrode holder in the vacuum tub of a vacuum evaporation system. This vacuum evaporation system is the existing thing, and opposite arrangement of a substrate electrode holder and the resistance heating boat made from two or more molybdenum is carried out into the vacuum tub. In being able to carry out the vacuum evaporation of the film of two or more layers continuously, without breaking the vacuum of a vacuum tub, it has come to be able to carry out the vacuum evaporation of the film which consists of two or more kinds of material in such a vacuum evaporation system by evaporating simultaneously the vacuum evaporation material from which a kind differs.

[0034] Subsequently, after putting the pellet for hole-injection layer 13 obtained by the processing mentioned above, the pellet for luminous layer 14 obtained by the same processing, and the metal for cathode 15 into a respectively different resistance heating boat, the inside of a vacuum tub is decompressed so that it may become a predetermined degree of vacuum. In addition, a powdered thing may be used for the metal for cathode 15, or a pellet-like thing may be used for it. Moreover, the kind of metal to be used is not limited to one kind, but it may be two or more kinds, and since vacuum evaporation conditions, such as vacuum evaporation temperature, differ for every quality of the material in using two or more kinds of metals, it is put into a respectively different resistance heating boat.

[0035] Then, to predetermined temperature, heat the resistance heating boat into which the pellet for hole-injection layer 13 was put, evaporate it, it is made to deposit on the anode plate 12 of the transparent support substrate 10, and the hole-injection layer 13 is formed. Since there is almost no absorption of the moisture after deaeration processing etc. while gas content is made one-mol% by

deaeration processing of the above-mentioned [the pellet for hole-injection layer 13] at this time, at the time of vacuum evaporatio, impurities, such as gas, are hardly generated from the pellet for hole-injection layer 13.

[0036] A luminous layer 14 is formed without taking out the transparent support substrate 10 from a vacuum tub, if membrane formation of the hole-injection layer 13 is completed. That is, to predetermined temperature, heat the resistance heating boat into which the pellet for luminous layer 14 was put, evaporate it, it is made to deposit on the hole-injection layer 13, and a luminous layer 14 is formed. Under the present circumstances, since the pellet for luminous layer 14 does not almost have absorption of the moisture after deaeration processing etc. like the case of the hole-injection layer 13 while gas content is made one-mol% by the above-mentioned deaeration processing, at the time of vacuum evaporatio, impurities, such as gas, are hardly generated from a pellet.

[0037] If membrane formation of a luminous layer 14 is completed, cathode 15 will be formed succeedingly. That is, to predetermined temperature, heat the resistance heating boat into which the metal for cathode was put, evaporate it, it is made to deposit on a luminous layer 14, and cathode 15 is formed. In addition, the cathode 15 of this operation gestalt is formed at intervals of [three] predetermined on a luminous layer 14 using a mask etc.

[0038] If the vacuum evaporatio of cathode 15 is completed, the transparent support substrate 10 which formed the hole-injection layer 13, a luminous layer 14, and cathode 15 will be taken out from a vacuum tub, it will cut to three, and lamination will obtain three organic EL elements 1 used as the 14/cathode 15 of 13/luminous layers of 12/(ITO film) hole-injection layers of anode plates.

[0039] According to such this operation gestalt, there are the following effects. That is, since the gas content is made into less than [1mol%] and each pellet (organic vacuum evaporatio material) which constitutes the hole-injection layer 13 and a luminous layer 14 can lessen sharply the amount of the gas as an impurity which evaporates at the time of vacuum evaporatio, it can reduce certainly contamination of each class 12-15 and the substrate 11 which constitute organic EL element 1 by gas. Moreover, since each pellet (organic vacuum evaporatio material) which constitutes the hole-injection layer 13 and a luminous layer 14 has an area smaller than a powdered thing in contact with the atmosphere and adsorption of the moisture which is an impurity is made to the minimum, evaporation of the moisture from the pellet at the time of the vacuum evaporatio of the hole-injection layer 13 and a luminous layer 14 etc. is made to the minimum, and contamination of the each class 12-15 and the substrate 11 which constitute organic EL element 1 by impurities, such as moisture, can be Since it is almost lost that each class 12-15 and the substrate 11 which constitute organic EL element 1 are polluted by gas, moisture, etc. by this at the time of vacuum evaporatio, low quality organic EL element 1 of driver voltage with a long life is obtained.

[0040] Furthermore, since each pellet of the hole-injection layer 13 and a luminous layer 14 was produced by heating organic substance powder in a vacuum, and gas, such as an organic solvent incorporated during the crystal of the organic substance, can also be removed and it can deaerate efficiently, the gas content in a pellet can be reduced certainly.

[0041] In addition, this invention is not limited to the aforementioned operation gestalt, and deformation as shown below etc. is included in this invention including other composition which can attain the purpose of this invention. That is, with the aforementioned operation gestalt, although pelletizing and deaeration processing of organic substance powder were performed simultaneously, after fabricating organic substance powder in the shape of a pellet by pressing etc., deaeration processing may be performed, or after carrying out deaeration processing of the organic substance powder, you may pelletize.

[0042] Although the lamination of an organic EL element explained the case of an anode plate / hole-injection layer / luminous layer / cathode with the operation gestalt described above, it is not limited to this, is good also as an anode plate / luminous layer / cathode, or is good also as an anode plate / luminous layer / electron-injection layer / cathode, and is still better also as an anode plate / hole-injection layer / luminous layer / electron-injection layer / cathode. Moreover, you may prepare a protective coat so that these layers may be covered.

[0043]

[Example] Next, a book

[Example 1] In this example 1, based on the aforementioned operation gestalt, each pellet (organic vacuum evaporatio material) which constitutes a hole-injection layer and a luminous layer was produced, and the organic EL element was produced using these pellets. The following concrete conditions etc. were adopted in this example 1.

[0044] <Pellet (organic vacuum evaporatio material)> ** raw material (organic substance powder)

- Hole-injection layer : powdered N, N'-diphenyl - N, N'-screw-(3-methylphenyl)-[I-I' biphenyl]-4, 4'-diamine (it is called Following TPD)

- Luminous layer : Powdered tris (eight quinolinol) aluminum (henceforth Alq3)

- The amount of supply to the container of each organic substance powder : 1g [0045]

** Vacuum evaporation system (the Japan vacuum-technology incorporated company make)

- Capacity of a vacuum tub : 650l. and exhaust air capacity : 104 The number of l / second ** container, and crevices : Heating of 10** organic-substance powder: After the degree of vacuum of a vacuum tub was set to 1×10 to 5 Pa, heating of a container was started, and it heated so that a container might become 200 degrees C. The degree of vacuum fell by generating of gas, and after returning to 1×10 to 5 Pa again, heating was continued for 10 more minutes.

[0046] As a result of performing fabrication and deaeration processing based on the above conditions etc. about the TPD powder which is organic substance powder for hole-injection layers, ten pellets of 100mg TPD were obtained. After the degree of vacuum of a vacuum tub once fell in 1×10 to 3 Pa and this state continued for 30 seconds by generating of gas at the time of heating of TPD powder, it returned to 1×10 to 5 Pa again. The fall of the degree of vacuum by heating at this time is about 1×10 to 3 Pa, and is this value and the exhaust speed 104 of a vacuum tub. If the capacity emitted from TPD powder is estimated by the equation of state for gases from l/second, about 4×10 to 6 mol/a second will come. Discharge of this gas is about 1×10^{-4} from TPD powder by deaeration processing according to heating of this example 1 since it continued for 30 seconds. It means that the gas of mol was emitted. The gas content of the rate for which gas accounts in 1g of used TPD powder by this, i.e., the TPD powder before heating, becomes about five-mol%. Moreover, during heating of TPD, since the degree of vacuum of a vacuum tub returned to 1×10 to 5 Pa, it is thought that the gas in TPD was emitted nearly completely. Therefore, it can be said that the gas content of the TPD pellet which is an organic vacuum evaporatio material is less than $[5 \times 10^{-3} \text{mol\%}]$.

[0047] Alq3 which similarly is the organic substance powder for luminous layers As a result of performing fabrication and deaeration processing about powder, it is 100mg Alq3. Ten pellets were obtained. Like the case of Above TPD, when capacity was calculated from change of the degree of vacuum of the vacuum tub under heating, it is Alq3 before heating. The powdered gas content was about eight-mol%. Moreover, Alq3 Alq3 as an organic vacuum evaporatio material since the degree of vacuum returned during heating at 1×10 to 5 Pa It can be said that the gas content of a pellet is less than $[5 \times 10^{-3} \text{mol\%}]$.

[0048]

<an organic EL element> -- composition [of ** element], and substrate : 60nm and, and luminous layer organic vacuum evaporatio material: -- aforementioned Alq3 pellet thickness : 60nm and cathode: -- it consists of a mixed metal of magnesium and silver Size : 1.1mm in 75mm[25mm by] x thickness, and anode plate Thickness : 10nm and hole-injection layer organic vacuum evaporatio material: -- the aforementioned TPD pellet Thickness

Vacuum evaporatio material ; Magnesium and silver Thickness ; 150nm Flat-surface size ; 3mmx15mm (one cathode)

- Luminescence side Flat-surface size : 3mmx5mm[0049]

** Vacuum evaporation system : Washing of the same thing ** transparent support substrate as what was used by production of an organic vacuum evaporatio material : It cleaned ultrasonically for 30 minutes by isopropyl alcohol. Pure water washes for 30 minutes the back, and it is isopropanal PIRUARU again. It cleaned ultrasonically for 30 minutes by the call.

** The pressure in the vacuum tub at the time of vacuum evaporation : 1×10^{-5} Pa [0050]
 ** Heating temperature of the boat into which the amount-used: 200 mg-Alq3 pellet which are the vacuum evaporation condition and a TPD pellet of a hole-injection layer was put : evaporation-rate : 0.1-0.2nm/second [0051] of 275 degree C and an Alq3 pellet : Heating temperature of the boat into which the 200 mg-TPD pellet was put: Evaporation rate of 215-220 degree C and a TPD pellet : The vacuum evaporation conditions of 0.1-0.3nm** luminous layer, Alq3 The amount of
 The vacuum evaporation conditions of cathode ** : Simultaneous [put magnesium and silver into a separate resistance heating boat and] The amount of - magnesium used been alike and evaporated : Heating temperature of the boat into which 1g and magnesium were put : The evaporation rate of about 500 degree C and magnesium : The amount of about 1.7-2.8nm/second and the silver used : Heating temperature of the resistance heating boat into which 500mg and silver were put : Evaporation rate of about 800 degree C and silver : 0.03-0.08nm [0052]/second According to the above conditions, based on the aforementioned operation gestalt, the vacuum evaporation of a hole-injection layer, a luminous layer, and the cathode was carried out one by one, and the organic EL element of this example 1 was obtained. The initial brightness of this organic EL element is voltage 6.5V and current density 3 mA/cm². It is 100 cd/m² under conditions. Reaching, the power conversion efficiency at this time was 1.6 lm/W.

[0053] [Example 1 of comparison] TPD powder and Alq3 which have not carried out deaeration processing by heating in the aforementioned example 1, respectively as an organic vacuum evaporation material of a hole-injection layer and a luminous layer The organic EL element of this example 1 of comparison was obtained like the aforementioned example 1 except having used powder. Thus, TPD and Alq3 If deaeration processing is omitted, the gas content of an organic vacuum evaporation material will increase more than one-mol%. For this reason, it is TPD and Alq3 in the case of the vacuum evaporation of a hole-injection layer and a luminous layer. Much gas was emitted rather than the case of the shell aforementioned example 1, and the degree of vacuum of a vacuum tub fell. The initial brightness of the organic EL element of this example 1 of comparison is voltage 7V and current density 3.5 mA/cm². It is 100 cd/m² under conditions. Reaching, the power conversion efficiency at this time was 1.3 lm/W.

[0054] [Evaluation (1) of an organic EL element] Aging of a reduction-by-half life and driver voltage was evaluated about each organic EL element obtained in the example 1 and the example 1 of comparison. These results are shown in drawing 2 and drawing 3 . Evaluation of a reduction-by-half life is initial brightness 300 cd/m² It carries out and is 150 cd/m² by fixed current drive. It carried out by finding the time taken to reach. Evaluation of driver voltage was performed by asking for voltage required in order to continue and acquire early current.

[0055] From drawing 2 , they are TPD powder and Alq3 at the aforementioned example 1. Since the hole-injection layer and the luminous layer were deposited using the pellet which carried out deaeration processing of the powder, respectively, and made the gas content less than [1mol%], generating of the gas under vacuum evaporation is almost lost. Therefore, it turns out that contamination of the element by gas can be reduced and the organic EL element of the outstanding quality with little elevation of the driver voltage accompanying time progress is obtained in the top where a reduction-by-half life is longer than the example 1 of comparison. On the other hand, at the example 1 of comparison, they are TPD powder and Alq3. Since powdered deaeration processing and powdered pelletizing are omitted, an element is polluted by the gas emitted at the time of vacuum evaporation, a reduction-by-half life becomes short rather than the organic EL element of an example 1, and also the power surge accompanying time progress is large, and a bird clapper is known.

[0056] [Example 2] In the aforementioned example 1, the organic EL element of this example 2 was obtained like the aforementioned example 1 except gas content having used the 0.1-mol TPD pellet it is [pellet] % as an organic vacuum evaporation material of a hole-injection layer.

[0057] [Example 3] In the aforementioned example 1, the organic EL element of this example 3 was obtained like the aforementioned example 1 except gas content having used the one-mol TPD pellet it is [pellet] % as an organic vacuum evaporation material of a hole-injection layer.

[0058] [Example 4] In the aforementioned example 1, the organic EL element of this example 4 was obtained like the aforementioned example 1 except having used the TPD pellet whose gas content is $5 \times 10^{-2} \text{mol\%}$ as an organic vacuum evaporatio material of a hole-injection layer.

[0059] [Example 2 of comparison] In the aforementioned example 1, the organic EL element of this example 2 of comparison was obtained like the aforementioned example 1 except gas content having used the five-mol TPD pellet it is [pellet] % as an organic vacuum evaporatio material of a hole-injection layer.

[0060] [Evaluation (2) of an organic EL element] The reduction-by-half life was searched for like evaluation (1) of the aforementioned organic EL element about each organic EL element obtained in examples 2, 3, and 4 and the example 2 of comparison. These results are shown in drawing 4 as relation between the gas content of a TPD pellet, and a reduction-by-half life. Drawing 4 shows that the quality organic long EL element of a reduction-by-half life is obtained, so that there are few gas contents of a TPD pellet.

[0061]

[Effect of the Invention] Since gas content of an organic vacuum evaporatio material was made into less than [1mol%] and the amount of the gas as an impurity which evaporates at the time of the vacuum evaporatio of an organic layer can be sharply lessened in forming an organic layer using an organic vacuum evaporatio material according to this invention as stated above, contamination by gas can be reduced certainly. Moreover, since an organic vacuum evaporatio material was formed in the shape of a pellet and area which contacts the atmosphere rather than a powdered thing can be made small, adsorption of the moisture which is an impurity is made to the minimum. Therefore, since evaporation of the moisture from organic substance vacuum evaporatio material etc. is made to the minimum, contamination by impurities, such as moisture, can be reduced certainly.

[0062] Furthermore, since the thing of the shape of a pellet which made gas content less than [1mol%] was used as an organic vacuum evaporatio material in having carried out the vacuum evaporatio of the organic vacuum evaporatio material on the substrate, and forming the organic layer of an organic EL element and contamination of an organic layer or a substrate can be reduced certainly, the low quality organic EL element of driver voltage with a long life is obtained.

[Translation done.]

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TECHNICAL FIELD

[The technical field to which invention belongs] this invention relates to the organic EL element equipped with the vacuum evaporation method which forms an organic layer using an organic vacuum evaporation material, the electrode formed on the substrate, and the organic layer in detail about the vacuum evaporation method and an organic EL element (organic electroluminescent element).

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PRIOR ART

Background of the Invention] In recent years, the organic EL element which is a luminescence device containing an organic layer attracts attention, and research is advanced towards the use to a display etc. Generally this organic EL element is equipped with organic layers, such as an anode plate (transparent electrode) and a luminous layer, and the structure which carried out the laminating of the cathode (counterelectrode) on the substrate. On the other hand, in the electrophotography copying machine of the Carlsson method, the organic photo conductor equipped with the photosensitive layer which consists of the organic substance as an electrophotography photo conductor is used. Membrane formation of the organic layer in these organic EL elements, electrophotography photo conductors, etc. is performed by the vacuum deposition method which an organic vacuum evaporation material which consists of the organic substance is heated [vacuum deposition method], and evaporates it. The powdered thing which carried out melting deaeration and ground the organic substance of a raw material is used for this organic vacuum evaporation material.

[0003] However, in the organic substance's tending to adsorb the gas which is an impurity at the time of the pulverization etc., since an organic powdered vacuum evaporation material has the large touch area with the atmosphere, it is easy to adsorb impurities, such as moisture. At the time of the vacuum evaporation of an organic layer, gas, moisture, etc. which were adsorbed evaporate with an organic vacuum evaporation material, and it mixes in an organic layer or they adhere to a substrate etc. Because of contamination by impurities, such as such gas, the target function may not fully be obtained in an organic EL element, an electrophotography photo conductor, etc. By using what carried out pressing of the organic substance powder, and was used as the tablet as what solves such a problem as an organic vacuum evaporation material, the method (JP,5-204196,A) of suppressing evaporation of the gas at the time of vacuum evaporation and an organic vacuum evaporation material made to evaporate are introduced into a cold trap, and after making an impurity condense and removing, the method (JP,50-3973,A) of using for vacuum evaporation etc. is proposed.

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EFFECT OF THE INVENTION

[Effect of the Invention] Since gas content of an organic vacuum evaporation material was made into less than [1mol%] and the amount of the gas as an impurity which evaporates at the time of the vacuum evaporation of an organic layer can be sharply lessened in forming an organic layer using an organic vacuum evaporation material according to this invention as stated above, contamination by gas can be reduced certainly. Moreover, since an organic vacuum evaporation material was formed in the shape of a pellet and area which contacts the atmosphere rather than a powdered thing can be made small, adsorption of the moisture which is an impurity is made to the minimum. Therefore, since evaporation of the moisture from organic substance vacuum evaporation material etc. is made to the minimum, contamination by impurities, such as moisture, can be reduced certainly.

[0062] Furthermore, since the thing of the shape of a pellet which made gas content less than [1mol%] was used as an organic vacuum evaporation material in having carried out the vacuum evaporation of the organic vacuum evaporation material on the substrate, and forming the organic layer of an organic EL element and contamination of an organic layer or a substrate can be reduced certainly, the low quality organic EL element of driver voltage with a long life is obtained.

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TECHNICAL PROBLEM

[Problem(s) to be Solved by the Invention] By the method of tablet-izing organic substance powder, since it is only only carrying out pressing of the organic substance powder, gas, the organic solvent especially incorporated during the crystal of the organic substance cannot be removed, but there is a problem that contamination by gas cannot fully be reduced out of vacuum evaporatio material. On the other hand, by the method of introducing into a cold trap, although the nonvolatile component of the impurities in an organic vacuum evaporatio material is removable, since it cannot fully remove volatile components, such as gas, it cannot avoid contamination by gas etc. Moreover, if an organic EL element is created by these methods, since contamination of a luminous layer, a substrate, etc. cannot fully be suppressed, element performance degradation and the problem that a life becomes short especially arise.

[0005] The purpose of this invention is in the vacuum evaporatio method which forms an organic layer using an organic vacuum evaporatio material to offer the vacuum evaporatio method that contamination by impurities, such as gas and moisture, can be reduced certainly. Moreover, other purposes of this invention are in the organic EL element equipped with the electrode and organic layer which were formed on the substrate to offer the organic EL element which can secure the quality which was excellent by reducing contamination by the impurity.

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MEANS

[Means for Solving the Problem] Although it was based also on the property of the gas contained in an organic vacuum evaporative material as a result of this invention persons' inquiring wholeheartedly in order to solve the aforementioned technical problem, when the gas beyond 5mol% was contained in an organic vacuum evaporative material, degasifying occurred from an organic vacuum evaporative material at the time of vacuum evaporation, and the knowledge that an organic layer, a substrate, etc. were polluted by this gas was acquired. In addition, gas here is matter which is below the vacuum evaporative temperature grade of an organic vacuum evaporative material, and evaporates, and the matter which evaporates at temperature higher than the temperature near vacuum evaporative temperature is not included. this invention is restricting the amount of the gas in an organic vacuum evaporative material, and tends to attain the aforementioned purpose.

[0007] Specifically, this invention is the vacuum evaporative method which forms an organic layer using an organic vacuum evaporative material, and the aforementioned organic vacuum evaporative material is characterized by being formed in the shape of a pellet, and making gas content into less than [1mol%].

[0008] Here, gas means the matter which evaporates among the impurities in an organic vacuum evaporative material at the temperature below near the temperature at the time of the vacuum evaporation of an organic vacuum evaporative material among matter other than an organic vacuum evaporative material which carries out vacuum evaporation that is, irrespective of the organic substance and an inorganic substance. What it is heated not only at a gaseous thing but at the time of vacuum evaporation, and is evaporated in the atmosphere (ordinary temperature) is contained in this gas.

[0009] In this invention, since gas content of an organic vacuum evaporative material is made into less than [1mol%] and the amount of the gas as an impurity which evaporates at the time of the vacuum evaporation of an organic layer can be lessened sharply, contamination by gas can be reduced certainly. Moreover, rather than a powdered thing, since it is formed in the shape of a pellet, since an organic vacuum evaporative material can make area in contact with the atmosphere small, adsorption of the moisture which is an impurity is made as for it to the minimum. Therefore, since evaporation of the moisture from organic substance vacuum evaporative material etc. is made to the minimum, contamination by impurities, such as moisture, can be reduced certainly. Moreover, since heat conduction of itself becomes good unlike powder, when a lot of material is treated, vacuum evaporative conditions are stable for a short time.

[0010] As for the aforementioned organic vacuum evaporative material, it is above desirable by heating in a vacuum to carry out deaeration processing. Thus, if the organic substance is heated in a vacuum, since gas, such as an organic solvent incorporated during the crystal of the organic substance, is also removable and it can deaerate efficiently, the gas content in an organic vacuum evaporative material can be reduced certainly.

[0011] Furthermore, the gas content of the aforementioned organic vacuum evaporative material is less than [0.1mol%] more preferably, and gas content has a desirable method of a low. Moreover, in the

case of vacuum evaporation, since gas occurs from the vacuum tub itself, effect sufficient also by using the low organic vacuum evaporation material of gas content rather than this on the basis of the gas which occurs from this vacuum tub is acquired. The method of presuming from change of the degree of vacuum in the case of deaeration processing as a measuring method of gas content, the gas-chromatography method, the method of searching for from the weight reduction before and behind deaeration processing, etc. are employable. Moreover, although not limited, since it has a possibility of becoming at the time of vacuum evaporation and decomposing into an elevated temperature when especially the kind of organic vacuum evaporation material has too large molecular weight, the smaller one of molecular weight is desirable, for example, it is 2000 or less molecular weight.

[0012] Moreover, although especially the configuration or size of a pellet are not limited, what it is easy to create is desirable. As a configuration of a pellet, the shape of a globular shape and a pillar, a rectangular parallelepiped configuration, etc. are employable, for example. Moreover, as for the size of a pellet, it is desirable that an overall diameter sets to 0.1-100mm.

[0013] On the other hand, this invention is the organic electroluminescent element equipped with the electrode and organic layer which were formed on the substrate, and it is characterized by forming the aforementioned organic layer by carrying out the vacuum evaporation of the organic vacuum evaporation material on the aforementioned substrate, and forming the aforementioned organic vacuum evaporation material in the shape of a pellet, and making gas content into less than [1mol%].

[0014] In this invention, since the thing of the shape of a pellet which made gas content less than [1mol%] is used as an organic vacuum evaporation material, gas, moisture, etc. which are an impurity in an organic vacuum evaporation material can be lessened and contamination of an organic layer or a substrate can be reduced certainly, the low quality organic EL element of driver voltage with a long life is obtained.

[0015] By the type in which the aforementioned organic EL element considers the substrate side as the ejection side of light, the thing of following the (1) - (4) is mentioned for the order of a laminating on a substrate front face as an example of the lamination of an organic EL element.

(1) An anode plate / luminous layer / cathode (2) anode plate / luminous layer / electron-injection layer / cathode (3) anode plate / hole-injection layer / luminous layer / cathode (4) anode plate / hole-injection layer / luminous layer / electron-injection layer / cathode [0016] Here, usually, although a luminous layer is formed of a kind or two or more sorts of organic luminescent material, it may be formed of mixture with an organic luminescent material, hole-injection material, and/or electron-injection material. Moreover, the protective layer for preventing permeation of the moisture to an element on it, as the element concerned is covered on the periphery of the element of lamination mentioned above may be prepared.

[0017] When making a substrate side into the ejection side of light, the aforementioned substrate consists of matter which gives high permeability (in general 80% or more) to luminescence (EL light) from an organic EL element at least, and, specifically, the tabular object, sheet-like object, or film-like object which consists of transparent glass, transparent plastics, a quartz, etc. can be used for it.

[0018] As a material of an anode plate, cathode, a luminous layer, a hole-injection layer, an electron-injection layer, and a protective layer, a well-known material can be used conventionally, respectively. For example, as an anode material, a metal with a large (for example, 4eV or more) work function, an alloy, electrical conductivity compounds, or such mixture are used preferably. As an example, it is metals, such as gold and nickel, and CuI, ITO and SnO₂. Dielectric transparent materials, such as ZnO, etc. are mentioned. Especially, the point of productivity or a controllability to ITO is desirable. Although the thickness of an anode plate is based also on material, it is usually selectable suitably within the limits of 10nm - 1 micrometer.

[0019] Moreover, as a cathode material, the small (for example, 4eV or less) metal of a work function, an alloy, electrical conductivity compounds, or such mixture are used preferably. As an example, rare earth metals, such as the alloy of sodium, a sodium potassium alloy, magnesium, a lithium, magnesium, and silver or a mixed metal, aluminum, aluminum/aluminum 2O₃, an indium, and an ytterbium, etc. are mentioned. Although the thickness of cathode is based also on material, it is usually selectable suitably

within the limits of 10nm - 1 micrometer. Also in any of an anode plate and cathode, below hundreds of ohms / ** of the sheet resistance are desirable. In addition, the size of the work function made into criteria in case an anode material and a cathode material are chosen is not limited to 4eV.

[0020] An organic vacuum evaporation material (organic luminescent material) of the luminous layer which is an organic layer While being able to pour in an electron hole from an anode plate or a hole-injection layer at the time of the luminous layer for organic EL elements, i.e., electric-field impression The pouring function in which an electron can be poured in from cathode or an electron-injection layer, What is necessary is just to be able to form the layer which has the transportation function to which the poured-in charge (an electron and an electron hole at least on the other hand) is moved by the force of electric field, the luminescence function to offer the place of the reunion of an electron and an electron hole and to tie this to luminescence, etc. As the example, a benzothiazole system, a benzimidazole system, The fluorescent brightener and metal chelate-ized oxy-NOIDO compound of systems, such as a benzo oxazole system A styryl benzenoid compound, a JISUCHIRIRU pyrazine derivative, a polyphenyl system compound, 12-phthaloperi non, 1, 4-diphenyl-1,3-butadiene, 1, 1, 4, and 4-tetraphenyl-1,3-butadiene, a NAFUTARU imide derivative, The metal complex of a perylene derivative, an OKISA diazole derivative, an aldazine derivative, a peeler JIRIN derivative, a cyclopentadiene derivative, a pyrrolo pyrrole derivative, a styryl amine derivative, a coumarin system compound, an aromatic dimethyldiyne compound, and an eight-quinolinol derivative etc. is mentioned. Although especially the thickness of a luminous layer is not limited, it is usually suitably chosen within the limits of 5nm - 5 micrometers.

[0021] An organic vacuum evaporation material (hole-injection material) of the hole-injection layer as an organic layer should just have the pouring nature of an electron hole, or electronic obstruction nature. As the example, a triazole derivative, an OKISA diazole derivative, An imidazole derivative, the poly aryl alkane derivative, a pyrazoline derivative, A pyrazolone derivative, a phenylenediamine derivative, an arylamine derivative, An amino substitution chalcone derivative, an oxazole derivative, a styryl anthracene derivative, full -- me -- non -- a derivative, a hydrazone derivative, a stilbene derivative, and a silazane derivative -- Conductive polymer oligomer, such as a polysilane system compound, an aniline system copolymer, and thiophene oligomer, a porphyrin compound, an aromatic tertiary-amine compound, a styryl amine compound, an aromatic JIMECHIRI DIN system compound, etc. are mentioned. Although especially hole-injection layer thickness is not limited, either, it is usually suitably chosen within the limits of 5nm - 5 micrometers. A hole-injection layer may be two or more layer structure which consists of a kind of the material mentioned above, or two sorts or more and which may be structure much more and consists of two or more layers of the same composition or different-species composition.

[0022] The electron-injection layer which is an organic layer that what is necessary is just to have the function to transmit the electron poured in from cathode to a luminous layer as an example of the material (electron-injection material) nitration full -- me -- non -- a derivative, an anthra quinodimethan derivative, and a diphenyl quinone derivative -- Heterocycle tetracarboxylic acid anhydrides, such as a thiopyran dioxide derivative and a naphthalene perylene, A carbodiimide, a deflection ORENIRIDEN methane derivative, an anthra quinodimethan derivative, The metal complex of an anthrone derivative, an OKISA diazole derivative, and an eight-quinolinol derivative, a metal free phthalocyanine and a metal phthalocyanine or the thing by which these ends are replaced with the alkyl group, the sulfone machine, etc., a JISUCHIRIRU pyrazine derivative, etc. are mentioned. Although especially electron-injection layer thickness is not limited, either, it is usually suitably chosen within the limits of 5nm - 5 micrometers. An electron-injection layer may be two or more layer structure which consists of a kind of the material mentioned above, or two sorts or more and which may be structure much more and consists of two or more layers of the same composition or different-species composition.

[0023] And the copolymer which is made to carry out copolymerization of the monomer mixture containing at least a tetrafluoroethylene and a kind of comonomer, and is obtained as an example of the material of a protective layer, The fluorine-containing copolymer which has a cyclic structure in a copolymerization principal chain, polyethylene, polypropylene, A polymethylmethacrylate, a polyimide,

a polyurea, poly tetrapod FUROORO ethylene, A polychlorotrifluoroethylene resin, poly dichlorodifluoroethene, The copolymer of a chlorotrifluoroethylene and dichlorodifluoroethene, The absorptivity matter of 1% or more of water absorptions, and the dampproof matter of 0.1% or less of water absorptions, Metals, such as In, Sn, Pb, Au, Cu, Ag, aluminum, Ti, and nickel, MgO, SiO, SiO₂ aluminum₂ O₃ GeO, NiO, CaO, BaO, and Fe₂ O₃ Y₂ O₃ TiO₂ etc. -- a metallic oxide and MgF₂ LiF, AlF₃, and CaF₂ etc. -- a metal fluoride etc. is mentioned

[0024] Moreover, it is not limited especially about the formation method of each class (an anode plate and cathode are included) except the organic layer which constitutes an organic EL element, either. As the formation methods, such as an anode plate and cathode, to the pan which can apply a vacuum deposition method, the spin coat method, the cast method, the sputtering method, the LB method, etc., for example about a protective layer A vacuum deposition method, the spin coat method, the sputtering method, the cast method, the MBE (molecular beam epitaxy) method, The cluster ion beam method, the ion play DINGU method, a plasma polymerization method (the RF excited ion play DINGU method), a reactive-sputtering method, a plasma CVD method, laser CVD, heat CVD, gas source CVD, etc. are employable.

[0025] Although the formation method of each class except an organic layer can be suitably changed according to the material to be used, when aiming at simplification of a facility, and shortening of the production time since an organic EL element can be formed only by this vacuum deposition method if a vacuum deposition method is used in formation of each class which constitutes an organic EL element, it is advantageous.

[0026] In addition, the configuration and size of each class (a protective layer is included) which constitute an organic EL element are not necessarily the same. Moreover, when plane view of the element is carried out, all other layers are not surely settled on the electrode formed in right above [of a substrate].

[0027]

[Embodiments of the Invention] Hereafter, one gestalt of operation of this invention is explained based on a drawing.

<Composition of an organic EL element> Organic EL element 1 of this operation gestalt is shown in drawing 1 . This organic EL element 1 is equipped with a substrate 11, the anode plate (transparent electrode) 12 which consists of an ITO film formed on this substrate 11, the hole-injection layer 13 as an organic layer and luminous layer 14 which were formed on this anode plate 12, and these organic layers 13 and the cathode (counterelectrode) 15 formed on 14.

[0028] A substrate 11 is a rectangle plate-like glass substrate, and the band-like anode plate 12 is formed by the one side along with the longitudinal direction. On the hole-injection layer 13 by which the laminating was carried out on this anode plate 12, and the luminous layer 14, three band-like cathode 15 prolonged in the direction which intersects perpendicularly with the longitudinal direction of a substrate 11, i.e., the direction which intersects perpendicularly with an anode plate 12, is formed. In organic EL element 1 of this operation gestalt, when plane view is carried out, an anode plate 12 and cathode 15 lap, and the luminescence side 16 of a flat-surface configuration rectangle is formed in the field to which the hole-injection layer 13 and the luminous layer 14 were infixed among these electrodes 12 and 15.

[0029] The hole-injection layer 13 which is an organic layer among such organic EL elements 1 is formed by carrying out the vacuum evaporation of the organic vacuum evaporation material for the hole-injection layer 13 concerned on the anode plate 12 of a substrate 11. The luminous layer 14 which is an organic layer is formed like this by carrying out the vacuum evaporation of the organic vacuum evaporation material for the luminous layer 14 concerned on the hole-injection layer 13 of a substrate 11. Such organic vacuum evaporation material is formed in the shape of a pellet, and the gas content is made into less than [1mol%].

[0030] <Production of an organic vacuum evaporation material> Each organic vacuum evaporation material which constitutes the hole-injection layer 13 and a luminous layer 14 is produced in the respectively following procedures. With this operation gestalt, deaeration processing and fabrication of the organic substance are performed by heating the powdered organic substance in a vacuum. That is,

organic substance powder is put into the container made from molybdenum, and it sets in the vacuum tub of a vacuum evaporation system. In addition, this container has two or more crevices of the configuration corresponding to the pellet, and puts the organic substance powder of the specified quantity into these crevices, respectively.

[0031] Subsequently, if a vacuum tub is exhausted with a vacuum pump and a vacuum tub becomes a predetermined degree of vacuum, a container will be heated so that it may become predetermined temperature. In addition, what is necessary is just to measure the degree of vacuum of a vacuum tub using an ionization vacuum gage etc. If a container is heated, while the organic substance powder in the crevice will fuse, the gas as an impurity contained in the organic substance is emitted, and, thereby, the degree of vacuum of a vacuum tub falls. If it continues heating a container to predetermined temperature as it is, the gas in organic substance powder will decrease, the evaporation will decrease, and, thereby, the degree of vacuum of a vacuum tub will become high. And if the gas contained in organic substance powder is almost exhausted, the degree of vacuum of a vacuum tub will return to the aforementioned predetermined degree of vacuum (degree of vacuum before heating). The gas in organic substance powder is removed by such deaeration processing. Gas, such as an organic solvent incorporated during the crystal of the organic substance, is certainly removable by heating organic substance powder especially. The heating temperature of the container at this time is set as the temperature which is a grade in which the organic substance fuses and does not evaporate, and the heating time of a container is set up more than the time taken to recover the degree of vacuum which fell by generating of gas to a predetermined degree of vacuum (degree of vacuum before heating).

[0032] After returning to the state before the degree of vacuum of a vacuum tub heating, heating of a container is ended, the fused organic substance is cooled, and since the configuration where the crevice of a container was imitated is solidified, it takes out from a vacuum tub. Thereby, the pellet which is an organic vacuum evaporatio material is obtained. Thus, organic substance powder is heated in a vacuum, and if it deaerates until it returns to the state before the degree of vacuum of a vacuum tub heating, the gas content of the pellet obtained will become less than [1mol%]. Moreover, since a pellet becomes smaller than the time of the surface area in contact with the atmosphere being in a powder state, absorption of the moisture after deaeration processing etc. can be suppressed to the minimum.

[0033] <Production of an organic EL element> With this operation gestalt, the hole-injection layer 13, a luminous layer 14, and cathode 15 are formed one by one by the vacuum deposition method to the substrate 11 with anode plate 12. That is, after preparing the transparent support substrate 10 which formed the ITO film 12 beforehand on a substrate 11 and washing this transparent support substrate 10, it fixes to the substrate electrode holder in the vacuum tub of a vacuum evaporation system. This vacuum evaporation system is the existing thing, and opposite arrangement of a substrate electrode holder and the resistance heating boat made from two or more molybdenum is carried out into the vacuum tub. In being able to carry out the vacuum evaporatio of the film of two or more layers continuously, without breaking the vacuum of a vacuum tub, it has come to be able to carry out the vacuum evaporatio of the film which consists of two or more kinds of material in such a vacuum evaporation system by evaporating simultaneously the vacuum evaporatio material from which a kind differs.

[0034] Subsequently, after putting the pellet for hole-injection layer 13 obtained by the processing mentioned above, the pellet for luminous layer 14 obtained by the same processing, and the metal for cathode 15 into a respectively different resistance heating boat, the inside of a vacuum tub is decompressed so that it may become a predetermined degree of vacuum. In addition, a powdered thing may be used for the metal for cathode 15, or a pellet-like thing may be used for it. Moreover, the kind of metal to be used is not limited to one kind, but it may be two or more kinds, and since vacuum evaporatio conditions, such as vacuum evaporatio temperature, differ for every quality of the material in using two or more kinds of metals, it is put into a respectively different resistance heating boat.

[0035] Then, to predetermined temperature, heat the resistance heating boat into which the pellet for hole-injection layer 13 was put, evaporate it, it is made to deposit on the anode plate 12 of the

transparent support substrate 10, and the hole-injection layer 13 is formed. Since there is almost no absorption of the moisture after deaeration processing etc. while gas content is made one-mol% by deaeration processing of the above-mentioned [the pellet for hole-injection layer 13] at this time, at the time of vacuum evaporatio, impurities, such as gas, are hardly generated from the pellet for hole-injection layer 13.

[0036] A luminous layer 14 is formed without taking out the transparent support substrate 10 from a vacuum tub, if membrane formation of the hole-injection layer 13 is completed. That is, to predetermined temperature, heat the resistance heating boat into which the pellet for luminous layer 14 was put, evaporate it, it is made to deposit on the hole-injection layer 13, and a luminous layer 14 is formed. Under the present circumstances, since the pellet for luminous layer 14 does not almost have absorption of the moisture after deaeration processing etc. like the case of the hole-injection layer 13 while gas content is made one-mol% by the above-mentioned deaeration processing, at the time of vacuum evaporatio, impurities, such as gas, are hardly generated from a pellet.

[0037] If membrane formation of a luminous layer 14 is completed, cathode 15 will be formed succeeding. That is, to predetermined temperature, heat the resistance heating boat into which the metal for cathode was put, evaporate it, it is made to deposit on a luminous layer 14, and cathode 15 is formed. In addition, the cathode 15 of this operation gestalt is formed at intervals of [three] predetermined on a luminous layer 14 using a mask etc.

[0038] If the vacuum evaporatio of cathode 15 is completed, the transparent support substrate 10 which formed the hole-injection layer 13, a luminous layer 14, and cathode 15 will be taken out from a vacuum tub, it will cut to three, and lamination will obtain three organic EL elements 1 used as the 14/cathode 15 of 13/luminous layers of 12/(ITO film) hole-injection layers of anode plates.

[0039] According to such this operation gestalt, there are the following effects. That is, since the gas content is made into less than [1mol%] and each pellet (organic vacuum evaporatio material) which constitutes the hole-injection layer 13 and a luminous layer 14 can lessen sharply the amount of the gas as an impurity which evaporates at the time of vacuum evaporatio, it can reduce certainly contamination of each class 12-15 and the substrate 11 which constitute organic EL element 1 by gas. Moreover, since each pellet (organic vacuum evaporatio material) which constitutes the hole-injection layer 13 and a luminous layer 14 has an area smaller than a powdered thing in contact with the atmosphere and adsorption of the moisture which is an impurity is made to the minimum, evaporation of the moisture from the pellet at the time of the vacuum evaporatio of the hole-injection layer 13 and a luminous layer 14 etc. is made to the minimum, and contamination of the each class 12-15 and the substrate 11 which constitute organic EL element 1 by impurities, such as moisture, can be Since it is almost lost that each class 12-15 and the substrate 11 which constitute organic EL element 1 are polluted by gas, moisture, etc. by this at the time of vacuum evaporatio, low quality organic EL element 1 of driver voltage with a long life is obtained.

[0040] Furthermore, since each pellet of the hole-injection layer 13 and a luminous layer 14 was produced by heating organic substance powder in a vacuum, and gas, such as an organic solvent incorporated during the crystal of the organic substance, can also be removed and it can deaerate efficiently, the gas content in a pellet can be reduced certainly.

[0041] In addition, this invention is not limited to the aforementioned operation gestalt, and deformation as shown below etc. is included in this invention including other composition which can attain the purpose of this invention. That is, with the aforementioned operation gestalt, although pelletizing and deaeration processing of organic substance powder were performed simultaneously, after fabricating organic substance powder in the shape of a pellet by pressing etc., deaeration processing may be performed, or after carrying out deaeration processing of the organic substance powder, you may pelletize.

[0042] Although the lamination of an organic EL element explained the case of an anode plate / hole-injection layer / luminous layer / cathode with the operation gestalt described above, it is not limited to this, is good also as an anode plate / luminous layer / cathode, or is good also as an anode plate / luminous layer / electron-injection layer / cathode, and is still better also as an anode plate / hole-

injection layer / luminous layer / electron-injection layer / cathode. Moreover, you may prepare a protective coat so that these layers may be covered.

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EXAMPLE

[Example] Next, a book

[Example 1] In this example 1, based on the aforementioned operation gestalt, each pellet (organic vacuum evaporatio material) which constitutes a hole-injection layer and a luminous layer was produced, and the organic EL element was produced using these pellets. The following concrete conditions etc. were adopted in this example 1.

[0044] <Pellet (organic vacuum evaporatio material)> ** raw material (organic substance powder)
- Hole-injection layer : powdered N, N'-diphenyl - N, N'-screw-(3-methylphenyl)-[I-I' biphenyl]-4, 4'-diamine (it is called Following TPD)

- Luminous layer : Powdered tris (eight quinolinol) aluminum (henceforth Alq3)

- The amount of supply to the container of each organic substance powder : 1g [0045]

** Vacuum evaporation system (the Japan vacuum-technology incorporated company make)

- Capacity of a vacuum tub : 650l. and exhaust air capacity : 104 The number of l / second ** container, and crevices : Heating of 10** organic-substance powder: After the degree of vacuum of a vacuum tub was set to 1×10 to 5 Pa, heating of a container was started, and it heated so that a container might become 200 degrees C. The degree of vacuum fell by generating of gas, and after returning to 1×10 to 5 Pa again, heating was continued for 10 more minutes.

[0046] As a result of performing fabrication and deaeration processing based on the above conditions etc. about the TPD powder which is organic substance powder for hole-injection layers, ten pellets of 100mg TPD were obtained. After the degree of vacuum of a vacuum tub once fell in 1×10 to 3 Pa and this state continued for 30 seconds by generating of gas at the time of heating of TPD powder, it returned to 1×10 to 5 Pa again. The fall of the degree of vacuum by heating at this time is about 1×10 to 3 Pa, and is this value and the exhaust speed 104 of a vacuum tub. If the capacity emitted from TPD powder is estimated by the equation of state for gases from l/second, about 4×10 to 6 mol/a second will come. Discharge of this gas is about 1×10^{-4} from TPD powder by deaeration processing according to heating of this example 1 since it continued for 30 seconds. It means that the gas of mol was emitted. The gas content of the rate for which gas accounts in 1g of used TPD powder by this, i.e., the TPD powder before heating, becomes about five-mol%. Moreover, during heating of TPD, since the degree of vacuum of a vacuum tub returned to 1×10 to 5 Pa, it is thought that the gas in TPD was emitted nearly completely. Therefore, it can be said that the gas content of the TPD pellet which is an organic vacuum evaporatio material is less than [5×10^{-3} mol%].

[0047] Alq3 which similarly is the organic substance powder for luminous layers As a result of performing fabrication and deaeration processing about powder, it is 100mg Alq3. Ten pellets were obtained. Like the case of Above TPD, when capacity was calculated from change of the degree of vacuum of the vacuum tub under heating, it is Alq3 before heating. The powdered gas content was about eight-mol%. Moreover, Alq3 Alq3 as an organic vacuum evaporatio material since the degree of vacuum returned during heating at 1×10 to 5 Pa It can be said that the gas content of a pellet is less than [5×10^{-3} mol%].

[0048]

<an organic EL element> -- composition [of ** element], and substrate : 60nm and, and luminous layer organic vacuum evaporatio material: -- aforementioned Alq3 pellet thickness : 60nm and cathode: -- it consists of a mixed metal of magnesium and silver Size : 1.1mm in 75mm[25mm by] x thickness, and anode plate Thickness : 10nm and hole-injection layer organic vacuum evaporatio material: -- the aforementioned TPD pellet Thickness

Vacuum evaporatio material ; Magnesium and silver Thickness ; 150nm Flat-surface size ; 3mmx15mm (one cathode)

- Luminescence side Flat-surface size : 3mmx5mm[0049]

** Vacuum evaporation system : Washing of the same thing ** transparent support substrate as what was used by production of an organic vacuum evaporatio material : It cleaned ultrasonically for 30 minutes by isopropyl alcohol. Pure water washes for 30 minutes the back, and it is isopropanol PIRUARU again. It cleaned ultrasonically for 30 minutes by the call.

** The pressure in the vacuum tub at the time of vacuum evaporatio : 1×10 to 5 Pa [0050]

** Heating temperature of the boat into which the amount-used:200 mg-Alq3 pellet which are the vacuum evaporatio condition and a TPD pellet of a hole-injection layer was put : evaporation-rate : 0.1-0.2nm/second [0051] of 275 degree C and an Alq3 pellet : Heating temperature of the boat into which the 200 mg-TPD pellet was put: Evaporation rate of 215-220 degree C and a TPD pellet : The vacuum evaporatio conditions of 0.1-0.3nm** luminous layer, Alq3 The amount of

The vacuum evaporatio conditions of cathode ** : Simultaneous [put magnesium and silver into a separate resistance heating boat and] The amount of - magnesium used been alike and evaporated : Heating temperature of the boat into which 1g and magnesium were put : The evaporation rate of about 500 degree C and magnesium : The amount of about 1.7-2.8nm/second and the silver used : Heating temperature of the resistance heating boat into which 500mg and silver were put : Evaporation rate of about 800 degree C and silver : 0.03-0.08nm [0052]/second According to the above conditions, based on the aforementioned operation form, the vacuum evaporatio of a hole-injection layer, a luminous layer, and the cathode was carried out one by one, and the organic EL element of this example 1 was obtained. The initial brightness of this organic EL element is voltage 6.5V and current density 3 mA/cm². It is 100 cd/m² under conditions. Reaching, the power conversion efficiency at this time was 1.6 lm/W.

[0053] [Example 1 of comparison] TPD powder and Alq3 which have not carried out deaeration processing by heating in the aforementioned example 1, respectively as an organic vacuum evaporatio material of a hole-injection layer and a luminous layer The organic EL element of this example 1 of comparison was obtained like the aforementioned example 1 except having used powder. Thus, TPD and Alq3 If deaeration processing is omitted, the gas content of an organic vacuum evaporatio material will increase more than one-mol%. For this reason, it is TPD and Alq3 in the case of the vacuum evaporatio of a hole-injection layer and a luminous layer. Much gas was emitted rather than the case of the shell aforementioned example 1, and the degree of vacuum of a vacuum tub fell. The initial brightness of the organic EL element of this example 1 of comparison is voltage 7V and current density 3.5 mA/cm². It is 100 cd/m² under conditions. Reaching, the power conversion efficiency at this time was 1.3 lm/W.

[0054] [Evaluation (1) of an organic EL element] Aging of a reduction-by-half life and driver voltage was evaluated about each organic EL element obtained in the example 1 and the example 1 of comparison. These results are shown in drawing 2 and drawing 3 . Evaluation of a reduction-by-half life is initial brightness 300 cd/m² It carries out and is 150 cd/m² by fixed current drive. It carried out by finding the time taken to reach. Evaluation of driver voltage was performed by asking for voltage required in order to continue and acquire early current.

[0055] From drawing 2 , they are TPD powder and Alq3 at the aforementioned example 1. Since the hole-injection layer and the luminous layer were deposited using the pellet which carried out deaeration processing of the powder, respectively, and made the gas content less than [1mol%], generating of the gas under vacuum evaporatio is almost lost. Therefore, it turns out that contamination of the element by gas can be reduced and the organic EL element of the outstanding quality with few rises of the driver voltage accompanying time progress is obtained in the top where a reduction-by-half life is longer than

the example 1 of comparison. On the other hand, at the example 1 of comparison, they are TPD powder and Alq3. Since powdered deaeration processing and powdered pelletizing are omitted, an element is polluted by the gas emitted at the time of vacuum evaporation, a reduction-by-half life becomes short rather than the organic EL element of an example 1, and also the power surge accompanying time progress is large, and a bird clapper is known.

[0056] [Example 2] In the aforementioned example 1, the organic EL element of this example 2 was obtained like the aforementioned example 1 except gas content having used the 0.1-mol TPD pellet it is [pellet] % as an organic vacuum evaporation material of a hole-injection layer.

[0057] [Example 3] In the aforementioned example 1, the organic EL element of this example 3 was obtained like the aforementioned example 1 except gas content having used the one-mol TPD pellet it is [pellet] % as an organic vacuum evaporation material of a hole-injection layer.

[0058] [Example 4] In the aforementioned example 1, the organic EL element of this example 4 was obtained like the aforementioned example 1 except having used the TPD pellet whose gas content is 5×10^{-2} mol% as an organic vacuum evaporation material of a hole-injection layer.

[0059] [Example 2 of comparison] In the aforementioned example 1, the organic EL element of this example 2 of comparison was obtained like the aforementioned example 1 except gas content having used the five-mol TPD pellet it is [pellet] % as an organic vacuum evaporation material of a hole-injection layer.

[0060] [Evaluation (2) of an organic EL element] The reduction-by-half life was searched for like evaluation (1) of the aforementioned organic EL element about each organic EL element obtained in examples 2, 3, and 4 and the example 2 of comparison. These results are shown in drawing 4 as relation between the gas content of a TPD pellet, and a reduction-by-half life. Drawing 4 shows that the quality organic long EL element of a reduction-by-half life is obtained, so that there are few gas contents of a TPD pellet.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] The cross section showing 1 operation gestalt of this invention.

[Drawing 2] The graph which shows the reduction-by-half life of each organic EL element of the example 1 of this invention, and the example 1 of comparison.

[Drawing 3] The graph which shows the power surge of each organic EL element of the aforementioned example 1 and the example 1 of comparison.

[Drawing 4] The graph which shows the gas content of TPD, and the reduction-by-half life of an organic EL element about the examples 2 and 3 and the example 2 of comparison of this invention.

[Description of Notations]

- 1 Organic Electroluminescent Element
- 10 Transparent Support Substrate
- 11 Substrate
- 12 Anode Plate
- 13 Hole-Injection Layer (Organic Layer)
- 14 Luminous Layer (Organic Layer)
- 15 Cathode
- 16 Luminescence Side

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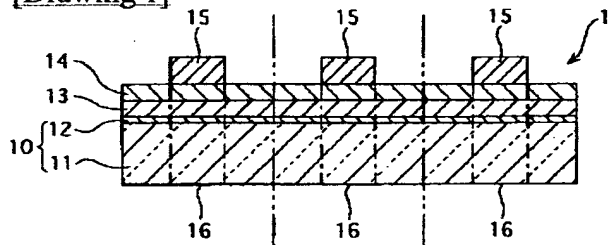
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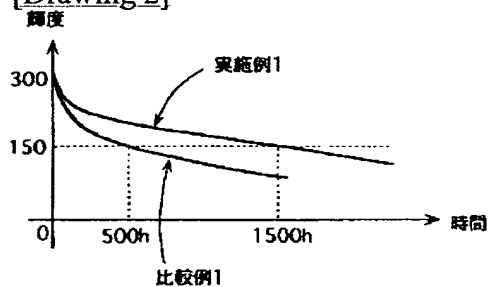
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DRAWINGS

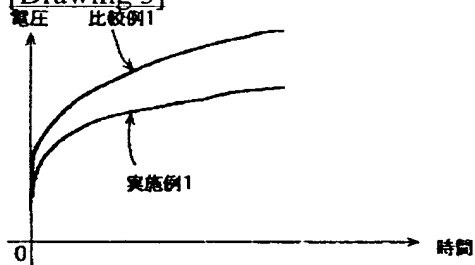
[Drawing 1]



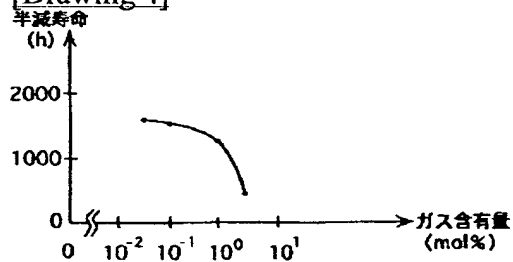
[Drawing 2]



[Drawing 3]



[Drawing 4]



[Translation done.]